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(54) Surface treatment method for translucent alumina ceramic

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## SPECIFICATION

### 1. TITLE OF THE INVENTION

Surface treatment method for translucent alumina ceramic

### 2. CLAIMS

What is claimed is:

1. A surface treatment method for translucent alumina ceramic wherein:

translucent alumina ceramic is heat-treated in a non-oxidative atmosphere or vacuum atmosphere at a temperature of between 1400°C and 1900°C after a grinding process and cutting process has been performed thereon.

### 3. DETAILED DESCRIPTION OF THE INVENTION

The present invention relates to a surface treatment method for translucent alumina ceramic, and in particular relates to a surface treatment method that smoothes the surfaces of transparent alumina ceramic that has a thin thickness by means of a heat treatment that is carried out on the ground and cut surfaces thereof.

Generally speaking, light scattering phenomena occur during the manufacture of translucent alumina ceramic due to the incorporation of gas pockets and impurities, as well as the size of the raw material particles and so forth. In particular, light scattering phenomena can be caused by surface unevenness due to the grinding process and cutting process, and there is a risk that this may result in a reduction

in optical transmittance.

By further polishing the ground and cut surfaces of the translucent alumina ceramic and providing them with a mirror finish, as is conventionally done, reduction in transmittance is prevented. However, due to the fact that translucent alumina ceramic is extremely hard, a large load must be placed thereon during the surface treatment by means of this type of mechanical polishing, and as a result, if the thickness of the alumina ceramic is thin, there is a risk that said load will damage it. If the surface treatment is carried out with a small load during polishing, damage to the alumina ceramic can be prevented; however, this lengthens the time needed for polishing, which causes a marked reduction in treatment efficiency.

The present invention resolves the disadvantage described above, and provides a surface treatment method for translucent alumina ceramic wherein the ground and cut translucent alumina ceramic can be made smooth in an extremely short amount of time, will not be damaged even if the thickness thereof is thin, and wherewith optical transmittance can be improved.

The present invention shall now be described in further detail.

A heat treatment is carried out on translucent alumina ceramic in a non-oxidative atmosphere (for example, in a hydrogen gas, nitrogen gas, or inert gas atmosphere) or vacuum atmosphere at a temperature of between 1400°C and 1900°C after a grinding process and cutting process has been carried out thereon, after which a treatment for smoothing the cut and ground surfaces of said translucent alumina ceramic is carried out.

The translucent alumina ceramic used in the present invention is typically that created by means of forming high-purity alumina particles by means of the isostatic press method, after which it is fired. The form of this transparent alumina ceramic can be sheet-like, tubular, or otherwise.

The reason for limiting the heat treatment temperature in the present invention to the range indicated above is because, if said heat

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treatment temperature is lower than 1400°C, adequate smoothing results cannot be achieved during the translucent alumina ceramic grinding process and cutting process. Conversely, if the temperature is higher than 1900°C, the translucent alumina ceramic deforms easily and dimensional accuracy is inhibited.

However, according to the present invention, by heat treating translucent alumina ceramic with ground and cut surfaces in a non-oxidative atmosphere or vacuum atmosphere at a prescribed temperature, said ground and cut surfaces can be grain-boundary diffused, surface diffused, and made into extremely smooth surfaces by means of the heat. Furthermore, impurities in the ground and cut surfaces or any other surfaces can be sublimated and removed by means of the heat treatment. Further, by carrying out the heat treatment in a non-oxidative atmosphere or vacuum atmosphere, the translucent alumina ceramic is prevented from turning yellow in color.

Consequently, translucent alumina ceramic with cut and ground surfaces (in particular that with a thin thickness) is not damaged, does not discolor, and is made smooth, while impurities on the surfaces thereof are removed, thus allowing for a marked improvement in the efficiency of the optical transmittance of the translucent alumina ceramic.

Working examples of the present invention shall now be described.

#### Working Example 1

First, a translucent alumina block with a density of 3.98g/cm<sup>3</sup> and particle diameter of 20-30 $\mu$  (dimensions: 12.5mm width  $\times$  150mm length  $\times$  12.5mm thickness) was cut into thin translucent alumina sheets 0.3mm thick using a 0.5t diamond cutter. Next, the cutting oil on these thin sheets was flushed with acetone, then with ion exchange water, after which the sheets were stacked onto a high-purity alumina ceramic sheet (99% Al<sub>2</sub>O<sub>3</sub>). They were then heat treated for 2 hours in a dry hydrogen gas atmosphere at 1600°C.

The surface-treated translucent alumina ceramic sheet (working example 1), non-heat-treated sheet (comparative example 1) and sheet provided with a mirror finish by means of polishing (comparative example 2) were then respectively irradiated with 250-350nm light using a Hitachi 424 spectrophotometer, and the optical transmittances thereof were measured in this wavelength range. The results are shown in the drawing. Line 1 is a curved line representing the optical transmittance in the thin sheet according to working example 1, line 2 is a curved line representing the optical transmittance in the thin sheet according to comparative example 1, and line 3 is a curved line representing the optical transmittance in the thin sheet according to comparative example 2.

What can be understood from the drawing is that the optical transmittance of the thin translucent alumina sheet treated by means of the method according to the present invention (line 1 in the drawing) was higher than the thin sheet on which heat treatment was not carried out (line 2 in the drawing). Further, the thin sheet treated by means of the method according to the present invention exhibited optical transmittance roughly equal to the thin sheet provided with a mirror finish (line 3 in the drawing), and in particular exhibited 90% optical transmittance with respect to light at 260nm.

The time required for surface treatment of the thin sheet treated by means of the conventional polishing method (comparative example 2) was 200 hours, this being extremely long in comparison to the 2 hours required for surface treatment according to the present invention and thus low in treatment efficiency.

#### Working Example 2

A translucent alumina tube with a density of 3.98g/cm<sup>3</sup> and a particle diameter of 50 $\mu$  (dimensions: 7.9 $\phi$  (ends)  $\times$  9.6 $\pm$ 0.2 $\phi$   $\times$  114mm length; diffuse transmittance: 94%) was ground to an external diameter of 9.6 $\pm$ 0.03 $\phi$  using a cylindrical grinding machine. The diffuse transmittance of this tube was 89%. Next, this tube was heat treated for 3 hours in a vacuum with 10<sup>-8</sup> torr or less at 1500°C.

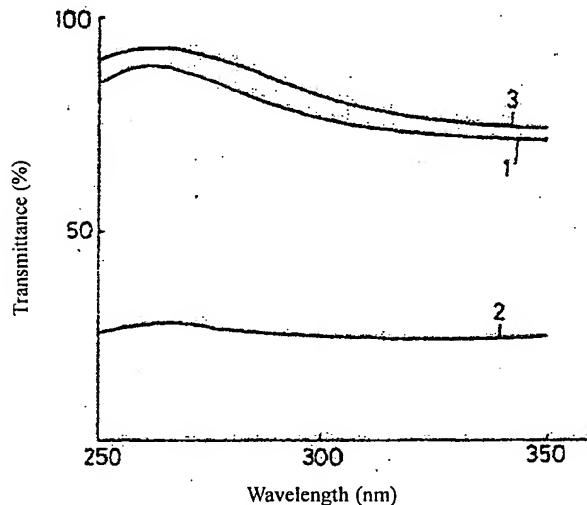
The diffuse transmittance of the obtained translucent alumina tube was measured with a PI-1 Tokyo Shibaura Electric spectrophotometer and was determined to be 94%, thus having restored transmittance to its pre-grinding level.

According to the present invention as described above, translucent alumina ceramic with cut and ground surfaces (in particular that with a thin thickness) can be made smooth in an extremely short amount of time without being damaged, while impurities on the surfaces thereof are removed, thus increasing the efficiency of the optical transmittance, this having marked effects for their use in arc tube bulbs, scale plates, etc.

Further, the method according to the present invention is also applicable to MgO, ZrO<sub>2</sub>, Y<sub>2</sub>O<sub>3</sub>, and other translucent ceramics besides translucent alumina ceramic.

#### 4. BRIEF DESCRIPTION OF THE DRAWING

The figure is a line drawing showing the optical transmittance of a thin translucent alumina ceramic sheet treated by means of the method according to the present invention, the same thin sheet with untreated cut and ground surfaces, and the same thin sheet whose cut and ground surfaces have been provided with a mirror finish.



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## ⑯透光性アルミナ磁器の表面処理方法

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## 明細書

## 1. 発明の名称

透光性アルミナ磁器の表面処理方法

## 2. 特許請求の範囲

透光性アルミナ磁器を研削加工ないし切断加工した後、非酸化性雰囲気もしくは真空雰囲気下で1400～1900℃の温度にて熱処理せしめることを特徴とする透光性アルミナ磁器の表面処理方法。

## 3. 発明の詳細な説明

本発明は透光性アルミナ磁器の表面処理方法に關し、詳しく述べては肉厚の薄い透光性アルミナ磁器の研削加工面、切断面を熱処理により平滑化する表面処理方法に係るものである。

一般に透光性アルミナ磁器は製造時における気孔、不純物の混入および原料粒子の大きさ等により光の散乱現象が起こるが、とくに製造後の研削加工ないし切断加工による表面の凹凸化により光の散乱現象を起こし、光透過率の低下を招く虞れがあつた。

しかし、従来は透光性アルミナ磁器の研削加工面ないし切断加工面を更に研磨して鏡面仕上げを行ない、透過率の低下を防止している。しかし、このような機械的研磨による表面処理方法にあつては、透光性アルミナ磁器が非常に硬いために、研磨時に大きな荷重を加えなければならず、その結果該アルミナ磁器が肉薄のものの場合、上記荷重によつて破損する虞れがある。しかるに、上記研磨時の荷重を小さくして処理すれば該アルミナ磁器の破損を抑制できるが、反面研磨時間が長くなり処理能率の著しい低下を招く欠点がある。

本発明は上記欠点を解消するためになされたもので、研削加工面ないし切断加工面を施した肉厚の薄い透光性アルミナ磁器でも破損することなく極めて短時間で平滑にでき、光透過率を改善できる透光性アルミナ磁器の表面処理方法を提供しようとするものである。

以下、本発明を詳細に説明する。

透光性アルミナ磁器を研削加工ないし切断加

工した後、非酸化性雰囲気（たとえば水素ガス、  
鷺素ガス、不活性ガス等の雰囲気）、もしくは真空雰囲気下で1400～1900℃の温度にて熱処理し、上記透光性アルミナ磁器の研削加工面ないし切断加工面を平滑に処理する。

本発明に使用する透光性アルミナ磁器は通常、高純度のアルミナ粒子をアイソスタティックプレス法により成形した後焼成せしめて造られる。この透光性アルミナ磁器の形状は板状、管状等任意である。

本発明における熱処理温度を上記範囲に限定した理由は、該熱処理温度を1400℃未満にすると、透光性アルミナ磁器の研削加工面ないし切断加工面を充分平滑化する効果が達成されず、一方その温度が1900℃を越えると、透光性アルミナ磁器が変形され易くなつて寸法精度が阻害されるからである。

しかして、本発明によれば研削加工面ないし切断加工面を有する透光性アルミナ磁器を非酸化性雰囲気もしくは真空雰囲気下で所定温度に

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アルミナ薄板を製作した。次いで、この薄板をアセトンで切断油を洗い流し、さらにイオン交換水で洗浄した後、これを高純度アルミナ磁器板（Al<sub>2</sub>O<sub>3</sub> 99%）上に積み重ね、乾燥水素ガス雰囲気中で1600℃の温度下にて2時間熱処理した。

しかして表面処理後の透光性アルミナ薄板（実施例1）、熱処理前の薄板（比較例1）および研磨により鏡面仕上げした薄板（比較例2）を、夫々分光光度計（日立製作所製：424型）を用いて250～350nmの光を照射し、該波長領域の光透過率を測定したところ、図の如き結果を得た。なお図中の実線1は実施例1の薄板における光透過曲線、実線2は比較例1の薄板における光透過曲線、実線3は比較例2の薄板における光透過曲線である。

図から明らかに本発明方法で処理された透光性アルミナ薄板（図中の実線1）は熱処理を施さない薄板（図中の実線2）に比して光透過率が著しく高いことがわかる。また本発明方

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熱処理することにより、上記研削加工面、切断加工面が熱によつて粒界拡散、表面拡散され、極めてその表面を平滑にできる。しかも上記熱処理により研削加工面、切断加工面およびその他の表面に存在する不純物を揮散除去できる。また、非酸化性雰囲気もしくは真空雰囲気下で熱処理することにより、透光性アルミナ磁器が黄色に着色されるのを防止できる。

したがつて、研削加工面ないし切断加工面を有する透光性アルミナ磁器（とくに薄物）を破損させず、かつ変色させずに平滑にできると同時にその表面に存在する不純物を除去できるため、透光性アルミナ磁器の光透過率を著しく効率よく改善できる。

次に、本発明の実施例を説明する。

#### 実施例1

まず、密度3.98g/cm<sup>3</sup>、結晶粒径20～30μの透光性アルミナブロック（寸法12.5mm×15.0mm×12.5mm）を、0.5tのダイヤモンドカッターにて肉厚0.3mmに切断して透光性

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法で処理された薄板は鏡面仕上げを施した薄板（図中の実線3）と略匹敵する光透過率を示し、とくに波長260nmの光に対し90%の光透過率を示した。

なお、本発明方法による薄板処理時間は2時間で行なえたのに対し、従来の研磨法による薄板（比較例2）の表面処理時間は200時間ときわめて長時間かかり、処理能率の低いものであつた。

#### 実施例2

密度3.98g/cm<sup>3</sup>、結晶粒径50μの透光性アルミナチューブ（寸法7.9mm（端部）×9.6±0.2mm×11.4mm、拡散透過率94%）を円筒研削盤を用いて外径9.6±0.03mmに研削加工した。このチューブの拡散透過率は89%であつた。次いで、このチューブを100℃トル以下での真空中で1500℃の温度下にて3時間熱処理した。

得られた透光性アルミナチューブを光電管照度計（東京芝浦電気製：P1-1型）により

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拡散透過率を測定したところ、その透過率は 94 %となり研削前の透過性を回復したことが判明された。

以上詳述した如く本発明によれば研削加工面ないし切削加工面を施した透光性アルミナ磁器（とくに内蔵のアルミナ磁器）を破損させるととなく極めて短時間で平滑になると同時にその表面に存在する不純物を除去して光透過率を効率よく向上でき、発光管バルブ、日盛板などに有効に利用できる等顕著な効果を有するものである。

なお、本発明方法は透光性アルミナ磁器の他、 $MgO$ 、 $ZrO_2$ 、 $Y_2O_3$ 、 $YAG$ 等の透光性磁器にも適用可能である。

#### 4 図面の簡単な説明

図は本発明方法により処理した透光性アルミナ薄板、切削加工面を処理していない同薄板および切削加工面を鏡面仕上げした同薄板の光透過率を示す線図である。

